SERDP / ESTCP Workshop

Surface Finishing & Repair Issues for Sustaining New Military Aircraft

U.S. AIR FORCE

Cadmium Alternatives for High-Strength Steel JTP – Phase II

Fiesta Resort and Conference Center
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Project Overview

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Objective

- Assess DoD-selected Cadmium alternatives in accordance with the DoD-approved Joint Test Protocol (JTP) for both traditional plating and brush plating of HSS applications.
  (JTP is available at www.jgpp.com – JCAT links)

Approach

- Three-phased approach:
  - Phase I (HE and adhesion testing): NAVAIR (complete)
  - Phase II (JTP test matrix): AFRL/CTC
  - Phase III (Testing for Threaded Fasteners): ARL
- Selection of candidates for further testing after each phase of testing is complete (Phases I & II)

Project Team Members

- **AFRL – Dr. Elizabeth Berman**
- ***CTC* – Mr. Neil Huber, Ms. Leanne Debias**
- **NAVAIR – Mr. Steve Brown**
- **Boeing – Mr. Joe Osborne**
- **ARL – Mr. Brian Placzankis**
- **WMTR – Mr. Jay Curry**
- **Hill AFB – Mr. Nate Hughes**
- **Alumiplate – Mr. Gus Vallejo**
- **Marshall Labs – Mr. John Marshall**

Phase I Overview and Selection Process

- **Alternatives Tested in Phase I**
  - Primary coatings:
    - Sputtered Aluminum (Marshall Labs)
    - Electroplated Aluminum (Alumiplate)
    - LHE Zn-Ni (Dipsol IZ-C17)
    - Acidic Zn-Ni (Boeing, Seattle)
    - Sn-Zn (Dipsol)
  - Repair coatings:
    - Brush Zn-Ni (SIFCO 4018)
    - Brush SN-ZN (LDC 5030)
    - Spray Aluminum-ceramic (Sermetel 249/273)
- **Tests Conducted**

  -Hydrogen Embrittlement
  -Re-embrittlement
  -Adhesion
- **Selection Process**

  -WebEx Teleconference to review results
  -E-mail voting to determine Phase II candidates

Alternative Selection – Phase I

Down-selected Coatings for Phase II:

- Primary test coatings
  - LHE Zinc-Nickel (Dipsol IZ-C17)
  - Electroplated Aluminum
  - Sputtered Aluminum
  - Controls:
    - Cadmium
    - IVD Al: Hill AFB flat panels
    - IVD Al: Cametoid fasteners, washers and HE bars
- Repair test coatings (Cd brush control)
  - Aluminum-Ceramic Repair Coating (Sermetel)
  - Zinc-nickel brush repair
  - Tin-Zinc brush repair
  - Control – Cd Brush

All Phase II testing methods will be performed according to the procedures and requirements in the JTP.

Phase II Tests

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Test Category	Test	Testing Facility
General Properties	Appearance	*CTC* (POC – Leanne Debias)
	Throwing power and alloy composition uniformity	*CTC*
	Strippability	NAVAIR (POC – Steve Brown)
	Galvanic potential	ARL (POC – Brian Plosankis)
Adhesion	Bend adhesion	NAVAIR
	Paint adhesion	NAVAIR
Corrosion	Unscribed NSS* (bare)	ARL
	Scribed NSS* (bare)	ARL
	Galvanic corrosion resistance	ARL
	Fluid corrosion resistance	ARL
	Scribed, painted salt spray	NAVAIR (paint), ARL (test)
	Scribed and unscribed SO_2 salt spray	NAVAIR
Lubricity	Run-on/Break-away torque	WMTR (POC – Jay Curry)
	Torque-tension & torque-tension of corrosion-exposed fasteners	WMTR
Reparability	Appearance & Thickness	*CTC*
	Bend adhesion	ARL
	Paint adhesion	ARL
	Scribed and unscribed salt spray	ARL
Quality Assurance	Hydrogen embrittlement – notched bar	NAVAIR

Primary Coating Test Summary

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Coating	Test				
	Appearance	Stripability	Bend Adhesion		
			4130 Steel	17-4 PH Stainless	Ti-6-4
LHE Cadmium (Baseline) – Hill AFB	Pass	N/A	N/A	N/A	N/A
IVD Aluminum (Baseline) – Hill AFB	Pass	N/A	N/A	N/A	N/A
IVD Aluminum (Baseline) – Cametoid Technologies	Pass	N/A	N/A	N/A	N/A
LHE Zinc-Nickel (Dipsol IZ-C17)	Pass	Fail	Pass	Pass	Fail
Electroplated Aluminum	Pass	Pass	Pass	Pass	Fail
Sputtered Aluminum	Pass	Pending	Pass	Pass	Pass

- **All primary coatings passed appearance testing**
- **Mixed stripability results**
  - **LHE Zinc-Ni failed due to change in bend adhesion in 1-2 bend cycles**
- **Mixed Bend Adhesion Results**
  - **All Primary Coatings passed using both 4130 steel & 17-4 stainless**
  - **LHE Zinc-Ni and electroplated Al failed bend adhesion for Ti-6-4 due to spalling and edge buckling respectively**

Primary Coating Appearance Test Results

Coating	Appearance Results
LHE Cadmium (Baseline) – Hill AFB	Coating is continuous but not uniform, showing some edge effect; coating is smooth, adherent, and free from blisters, pits, excessive powder, and contamination
IVD Aluminum (Baseline) – Hill AFB	Coating is continuous, uniform, smooth, adherent, and free from blisters, pits, excessive powder, and contamination
IVD Aluminum (Baseline) – Cametoid Technologies	Coating is continuous, uniform, smooth, adherent, and free from blisters, pits, excessive powder, and contamination
LHE Zinc-Nickel (Dipsol IZ-C17)	Coating is continuous but not uniform also containing a few spots of possible contamination; otherwise, the coating is smooth, adherent, and free from pits, blisters, and excessive powder
Electroplated Aluminum	Coating is continuous, uniform, smooth, adherent, and free from blisters, pits, excessive powder, and contamination
Sputtered Aluminum	Coating is continuous, uniform, smooth, adherent, and free from blisters, pits, excessive powder, and contamination

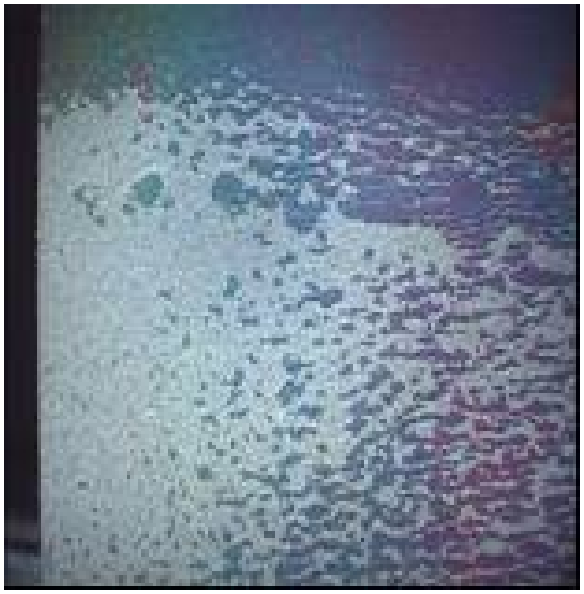
Primary Coating Stripability Test Results

Coating	Change in Hydrogen Embrittlement	Change in Bend Adhesion
LHE Cadmium (Baseline) – Hill AFB	Not required	Not required
IVD Aluminum (Baseline) – Hill AFB	Not required	Not required
IVD Aluminum (Baseline) – Cametoid Technologies	Not required	Not required
LHE Zinc-Nickel (Dipsol IZ-C17)	Pass – average of 88.5% fracture strength for 200 hours (3 of 4 specimens)	Fail – coating failure in 1-2 bend cycles (Rework procedures?)
Electroplated Aluminum	Pass – average of 93.6% fracture strength for 200 hours (4 of 4 specimens)	Pass – no coating failure before substrate rupture (12 cycles)
Sputtered Aluminum	Pending	Pending

Primary Coating Bend Adhesion Test Results

Coating	Bend Adhesion Results		
	4130 steel substrate	17-4 PH stainless substrate	Ti-6-4 substrate
LHE Cadmium (Baseline) – Hill AFB	Not required	Pass	Pass
IVD Aluminum (Baseline) – Hill AFB	Not required	Not required	Not required
LHE Zinc-Nickel (Dipsol IZ-C17)	Pass – cracking of coating up to 3/8”	Pass – no cracking or defect	Fail – during 1st bend cycle; spalling beyond 3/8”
Electroplated Aluminum	Pass	Pass - no cracking or defect	Fail – edge buckling to ½”
Sputtered Aluminum	Pass- no cracking or defect	Pass - no cracking or defect	Pass - no cracking or defect

Primary Coating
Bend Adhesion Test Results



Zinc-Nickel on Ti-6-4

Electroplated
Aluminum on Ti-6-4

Primary Coating Test Summary (continued)

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Coating	Test					
	Paint Adhesion (1, 4, 7 day duration)			Run On / Breakaway Torque		Hydrogen Embrittlement
	MIL-PRF-23377, Class C2	MIL-PRF-85582, Class C1	MIL-PRF-85582, Class N	3/8" fastener	5/8" fastener	
LHE Cadmium (Baseline) – Hill AFB	Pending	Pending	Pending	N/A	discussion required	Pass
IVD Aluminum (Baseline) – Hill AFB	Pass all durations	Pass all durations	Pass all durations	N/A	discussion required	N/A
IVD Aluminum (Baseline) – Cametoid Technologies	N/A	N/A	N/A	Pending	discussion required	Pending
LHE Zinc-Nickel (Dipsol IZ-C17)	Pass all durations	Fail 4 & 7 day duration	Pass all durations	Pass	discussion required	Pending (Test performed in DI & salt water)
Electroplated Aluminum	Pass all durations	Pass all durations	Pass all durations	Pass	discussion required	Pass
Sputtered Aluminum	Pass all durations	Pass all durations	Pass all durations	Pass	discussion required	Pass

- **Primary Coatings Passed Paint Adhesion Testing with one exception (LHE Zinc-Ni failed for 85582 Class C1 after 4 and 7 days)**
- **Run On/Breakaway torque test results:**
  - **Passed for 3/8” fastener**
  - **5/8” fastener need to be discussed**

Primary Coating
Paint Adhesion (Wet Tape) Test Results

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Primer Application →	**MIL-PRF-23377, Class C2**			**MIL-PRF-85582, Class C1**			**MIL-PRF-85582, Class N**		
Test Duration →	**1 day**	**4 days**	**7 days**	**1 day**	**4 days**	**7 days**	**1 day**	**4 days**	**7 days**
Coating	**Test Results (average of 12 measurements)**								
IVD Aluminum (Hill AFB)	5B	5B	5B	5B	5B	5B	5B	5B	5B
LHE Zinc-Nickel (Dipsol IZ-C17)	5B	4B	4B	5B	1B	1B	5B	5B	5B
Electroplated Aluminum	5B	5B	5B	5B	5B	5B	5B	5B	5B
Sputtered Aluminum	5B	5B	5B	5B	5B	5B	5B	5B	5B

Primary Coating
Paint Adhesion (Wet Tape) Test Results

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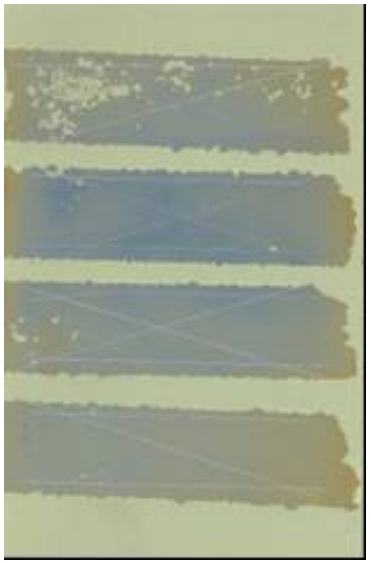
IVD Al with MIL-PRF-85582 C1, after 4 days



LHE Zn-Ni with MIL-PRF-85582 C1, after 4 days

Electroplated Al with MIL-PRF-23377, after 7 days

Sputtered Al with MIL-PRF-23377, after 4 days

Primary Coating Run-on/Breakaway Torque Test Results

- Reporting Maximum Locking Torque and Breakaway Torque for the 3/8-inch and 5/8-inch fasteners
- 3/8-inch LHE Zinc-Nickel, electroplated aluminum and sputtered aluminum fasteners pass JTP criteria (9.5 in-lbs)
- 5/8-inch Cadmium baseline and all coatings fail JTP criteria (32 in-lbs)
- Testing of the IVD Al fasteners coated by Cametoid Technologies is in progress at WMTR

Primary Coating Run-on/Breakaway Torque Test Results

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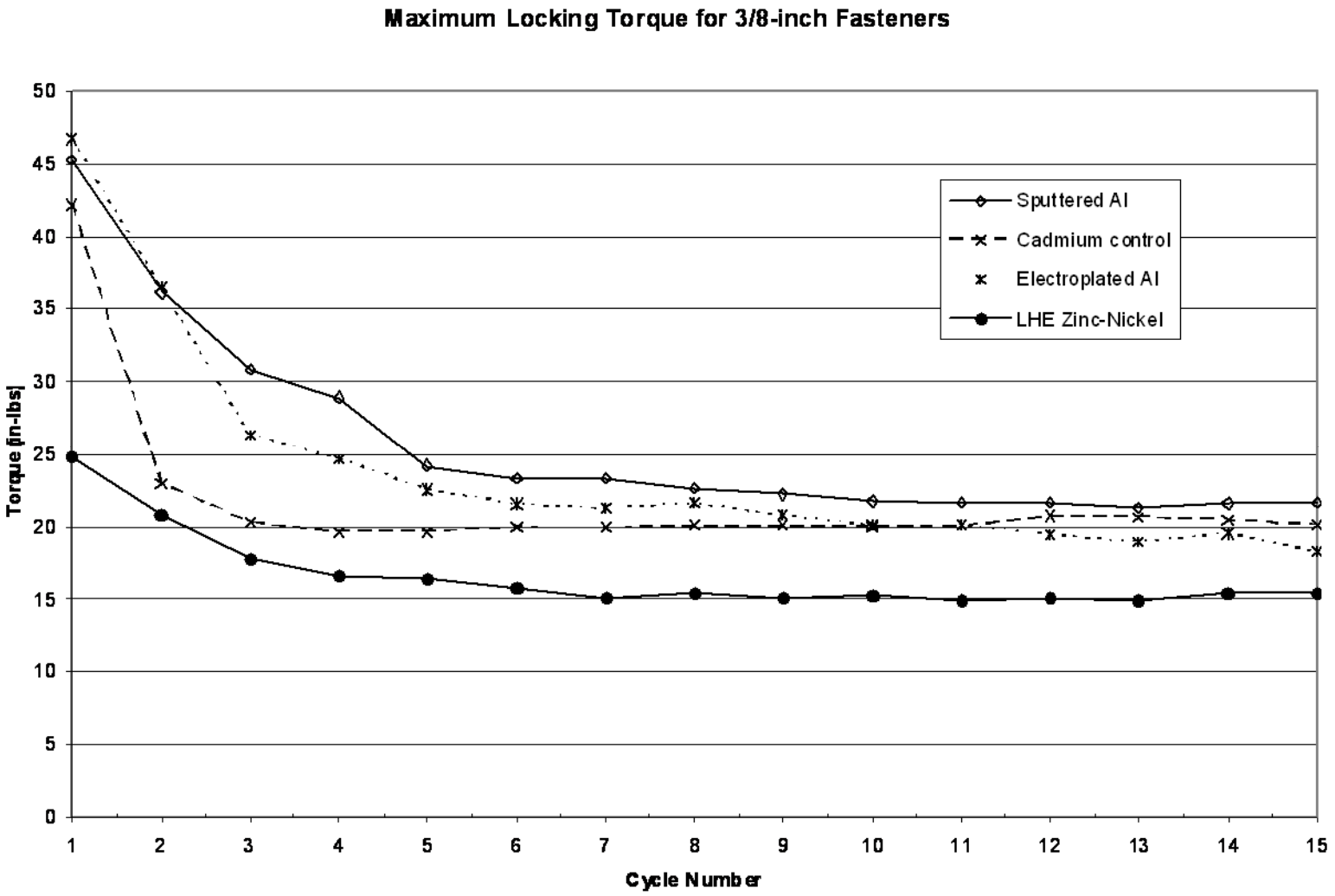

Primary Coating Run-on/Breakaway Torque Test Results

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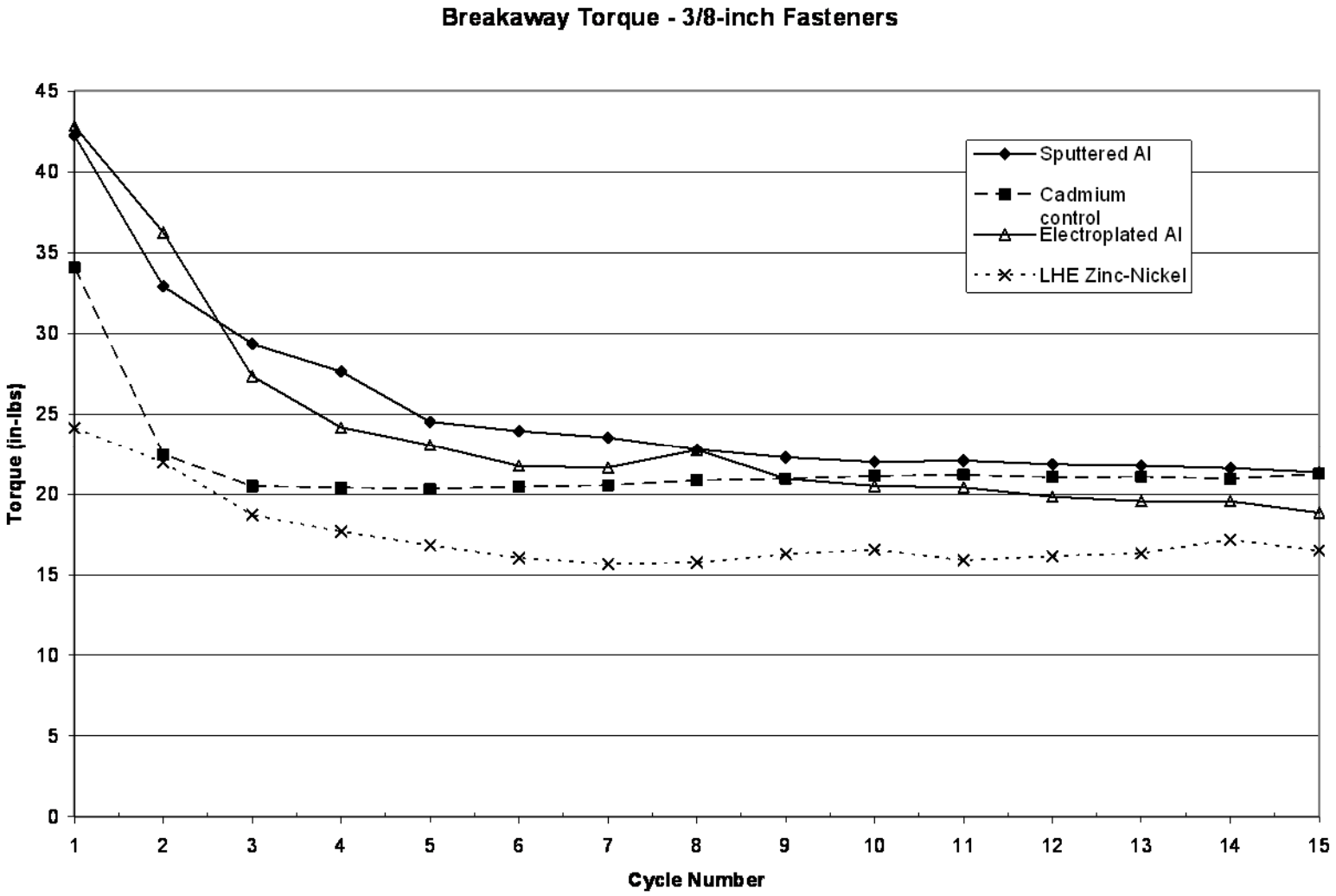

Primary Coating Run-on/Breakaway Torque Test Results

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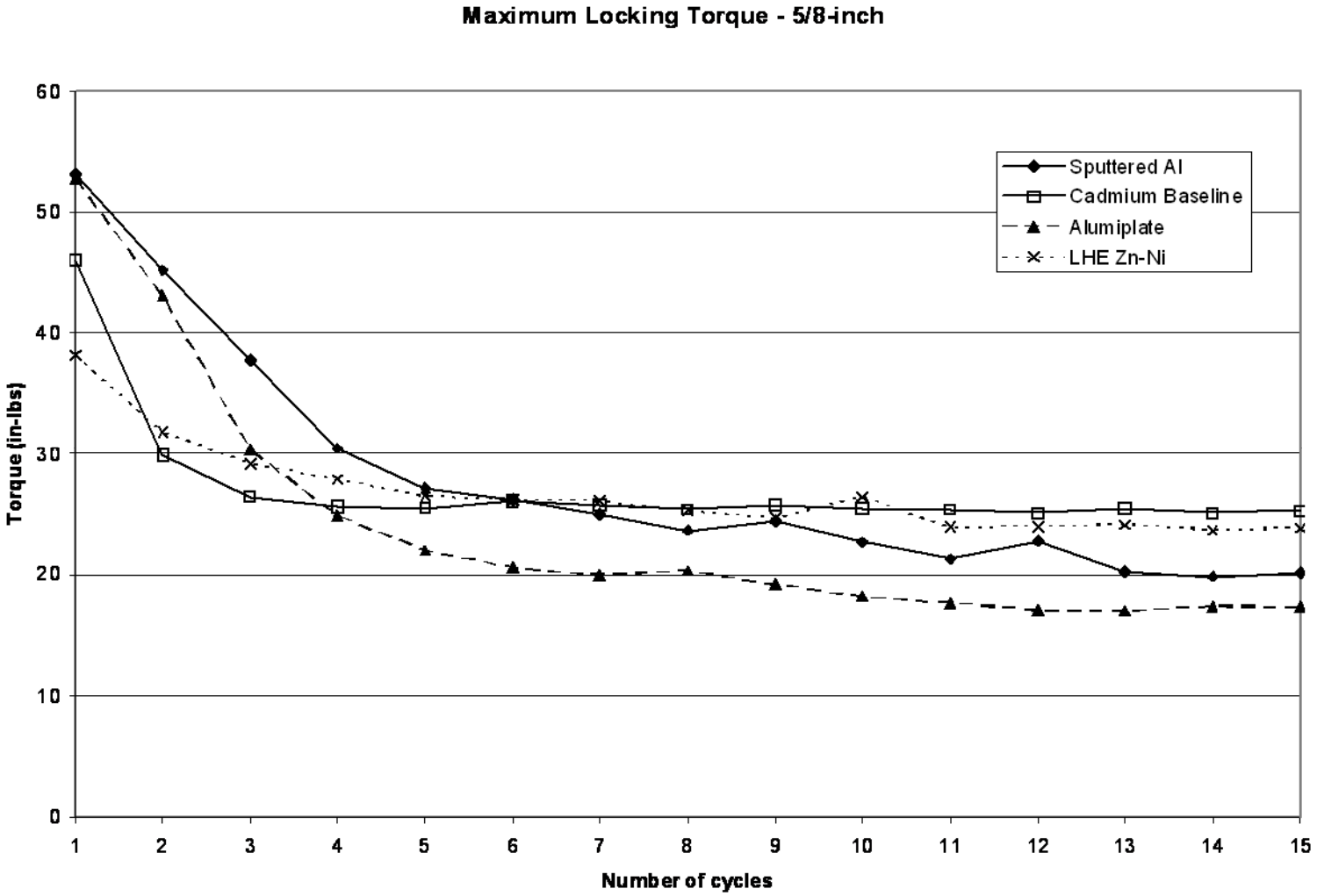
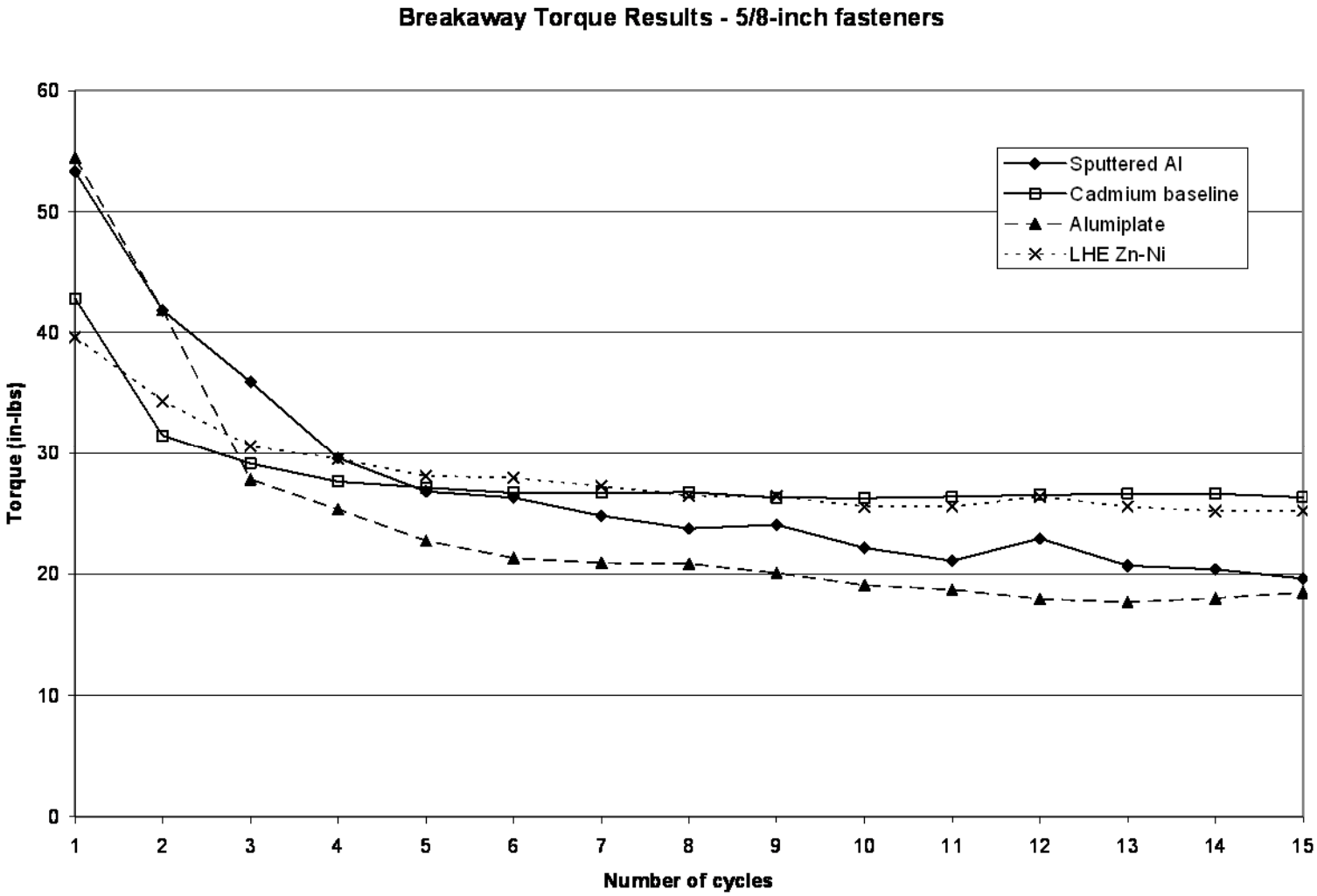


Primary Coating Run-on/Breakaway Torque Test Results



Repair Coating Test Summary

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Coating	Test	
	Appearance	Hydrogen Embrittlement
Brush LHE Cadmium – Boeing, St. Louis	Pass	Pass
Brush Tin-Zinc – Boeing, St. Louis	Fail	Pass
Brush Zinc-Nickel – Boeing, St. Louis	Pass	Pass
Sermetel 249/273 – Boeing, St. Louis	Pass	Pass

- Three of four Repair Coatings passed Appearance Testing
  - Brush Tin-Zinc failed, coating is not continuous or uniform; coating is adherent, but rough, with excessive powder and possible rust spots
- All Repair Coatings Passed Hydrogen Embrittlement testing

Repair Coating Appearance Test Results

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Coating	Appearance Results
Brush Plated Cadmium (Baseline) – Boeing, St. Louis	Coating is continuous but not uniform, showing swirls from processing; coating is smooth, adherent, and free from blisters, pits, excessive powder, and contamination
Brush Plated Zinc-Nickel – Boeing, St. Louis	Coating is not continuous or uniform; coating is adherent, but rough, with excessive powder and possible rust spots
Brush Plated Tin-Zinc – Boeing, St. Louis	Coating is continuous but not uniform, with a dark brown area through the center of the panel; the coating is smooth and adherent, but has excessive powder
Sermetel 249/273– Boeing, St. Louis	Coating is continuous and uniform, smooth, adherent, and free from pits, blisters, excessive powder, and contamination

Primary / Repair Coating Hydrogen Embrittlement Quality Assurance

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Coating	Average Fracture Strength (%)	Time to Failure (200 hour test)	Pass/Fail rating
LHE Cadmium (baseline) – Hill AFB	92.7%	204 hours	Pass
IVD Aluminum – Cametoid Technologies	Pending	Pending	Pending
Sputtered Aluminum – Marshall Labs	97.1%	204 hours	Pass
LHE Zinc-Nickel (Dipsol IZ-C17)	Pending	Pending	Pending
Electroplated Aluminum – Alumiplate	Pending	Pending	Pending
Brush LHE Cadmium – Boeing, St. Louis	96.3%	204 hours	Pass
Brush Tin-Zinc – Boeing, St. Louis	98.1%	205 hours	Pass
Brush Zinc-Nickel – Boeing, St. Louis	93.8%	203 hours	Pass
Sermetel 249/273 – Boeing, St. Louis	96.0%	204 hours	Pass

Anticipated Completion Dates for Testing in Progress

Test	Coating Type	Anticipated Completion Date
Throwing Power	Primary	Jan-08
Galvanic Potential	Primary	Mar-08
Neutral Salt Fog (bare)	Primary	Apr-08
Galvanic Corrosion	Primary	Mar-08
Fluid Corrosion	Primary	Mar-08
Neutral Salt Fog (painted)	Primary	May-08
Torque Tension	Primary	Feb-08
Bend Adhesion	Repair	Mar-08
Paint Adhesion	Repair	Mar-08
Neutral Salt Fog (bare)	Repair	Mar-08

Phase II Close-Out Process

- **Test Report will be issued for AFRL/JCAT review**
- ***CTC* will host a WebEx teleconference to review the Phase II Test Report**
- **Based on the discussion from the teleconference and the results of the Phase II testing, promising coating candidates for will be selected**
  - Any alternatives with clear deficiencies in a number of test categories will be eliminated from consideration

Timeline

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Activity	Start Date	End Date
Selection of Alternatives from Phase I	Nov-05	Jan-06
Formulation of Phases II and III Test Plan	Jan-06	Mar-06
Purchase/receive materials & secure subcons for Phase II	Mar-06	Sept-06
Ship and coat samples for Phase II	Sept-06	Dec-07
Phase II Testing	Nov-06	May-08
Phase II Final Report	Feb-07	Jun-08
Team Selection of Coating Alternatives - Phase II	Jun-08	Jul-08

Summary

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- Selected alternatives from Phase I have been coated
- Alternatives are currently in Phase II testing (*CTC*, NAVAIR, ARL, WMTR)
  - **~50% of tests complete with remainder to finish in 1st Qtr 08**
- Coating results to date show promise:
  - **Primary Coatings – 2 of 3 looks good**
    - **Electroplated and Sputtered Al have passed all 6 tests completed so far with one exception (Electroplated Al failed bend adhesion for Ti-6-4 but passed for 4130 Steel and 17-4 PH Stainless)**
    - **LHE Zinc-Ni alternative primary coating failed 3 of 6 tests completed so far (stripability, bend adhesion and paint adhesion)**
  - **Repair coatings also look good.**
    - **All have passed testing to date (appearance and HE) with exception of Brush Tin-Zinc failing appearance**
- The Phase II Final Report is planned to be complete in June 08
- JCAT will meet via teleconference to recommend best coating candidates from this Phase II effort. This is planned for June 08

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Back-Up Slides

Description of Testing Methods

- **General properties (primary coatings)**
  - Appearance – visual exam
  - Throwing power
    - Test fixture surrounds panel, with one access slot
    - Fixture + panel is placed in solution at 3 different orientations
    - Uniformity of coating is measured at 3 locations on each panel
  - Strippability
    - Specimens are stripped by vendor-recommended method
    - Half of specimens are tested
    - Remaining specimens are recoated and tested
      - Hydrogen Embrittlement
      - Adhesion
  - Galvanic Potential
    - Three types of measurements are performed over 5 days: open circuit potential measurement, electrochemical impedance spectroscopy, and tafel analysis

Description of Testing Methods (continued)

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- **Adhesion (primary coatings)**
  - Bend adhesion
    - Specimen is bent back and forth through 180° until the coating and/or substrate ruptures
  - Wet tape paint adhesion
    - Primers are applied to test panels (14 day cure)
      - MIL-PRF-85582 Type I, Class C1
      - MIL-PRF-85582, Type I, Class N
      - MIL-PRF-23377 Type 1, Class C
    - Panels are immersed in distilled water at following conditions:
      - 23°C for 24 hours
      - 49°C for 96 hours
      - 65°C for 168 hours
    - Perform tape adhesion according to ASTM D3359, Method A

Description of Testing Methods (continued)

- **Corrosion (primary coatings)**
  - Unscribed and Scribed Neutral Salt Spray (bare)
    - Bare panels exposed to a 5% NaCl solution sprayed at 35°C, until coating failure
  - Galvanic corrosion resistance
    - Components of test assemblies: 2024 or 7075 Al test block, coated with MIL-PRF-85582, Class 1, Type N, test washer (4 alloys), nuts, bolts, anodized washers
    - Test assemblies are exposed to salt fog for 168 hours and cyclic corrosion for 336 hours
  - Fluid corrosion resistance
    - Immerse panels in specified fluid at 100°F for 7 days
    - Test fluids: sea water, deicers, paint removers, cleaners, lubricants (14 total)

Description of Testing Methods (continued)

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- **Corrosion (continued)**
  - Scribed Painted Neutral Salt Spray
    - Test panels are primed with
      - MIL-PRF-85582 Type I, Class C1
      - MIL-PRF-85582, Type I, Class N
      - MIL-PRF-23377 Type 1, Class C
    - Test panels are exposed to 5% NaCl solution at 35°C for 3000 hours or until red rust
  - Scribed and Unscribed SO_2 Salt Spray
    - Unpainted panels and scribed, painted panels (same primers as above)
    - Expose to 5% NaCl and SO_2 gas IAW ASTM G85 A4 until coating failure (red rust)

Description of Testing Methods (continued)

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- **Lubricity (primary coatings)**
  - Run-on/Breakaway Torque
    - Record maximum locking torque after 2 complete turns from point where the top of the nut is flush with the end of the bolt
    - Breakaway torque is measured during removal of the nut
    - Measure for 15 lock/breakaway cycles and examine at 10x for thread damage
  - Torque Tension
    - Measure torque and induced load with test fixture for the range of 30%-60% of the bolt UTS
    - Repeat for a total of 5 cycles
  - Torque Tension of corrosion-exposed fasteners
    - Assemble bolts/nuts/washers onto an Al test block
    - Torque to 60% of UTS for bolt and exposed to cyclic corrosion for 28 days
    - Measure breakaway torque and compare to unexposed set

Description of Testing Methods (continued)

- **Repairability (repair brush coatings)**
  - Initial qualification – coating applied to bare substrate and tested
  - Final qualification – candidate primary coating of choice is abraded to generate a bare area and then repaired with a brush plating
  - Testing Methods
    - **Appearance** – visual exam
    - **Bend adhesion** – bend specimen back over itself until rupture
    - **Thickness** – cross-section and microscopy
    - **Scribed and unscribed salt spray (bare)** – until failure
    - **Paint adhesion** – apply primers, immerse in distilled water at same temps/times as primary coatings, and perform cross-hatch adhesion according to ASTM D3359, method B
- **Quality assurance – HE testing to compare to Phase I**